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Microwave-Assisted Preparation of Polyferric Sulfate Coagulant

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ABSTRACT

This study prepared polyferric sulfate (PFS) coagulants using a closed microwave method. In addition to reducing the requirement of adding oxidant and catalyst by applying the high pressure and high temperatures characteristic of microwave radiation, this work also adopts a newly developed continuous-flow process instead of the conventional batch production method to reduce the reaction time and prevent the energy waste. Water treatment plants can produce coagulants by themselves to meet their requirements since polyferric sulfate is easily created using this microwave method. The transport and storage expenses associated with providing reliable and fresh coagulants for water treatment can thus be reduced or eliminated. The approach specified herein has already been successfully applied to synthesize PFS products and evaluate products' physical and chemical properties, including ORP values, viscosity,

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salicylic acid reaction ability, and hydrolysis parameter B^*. The results reveal that PFS made using the microwave method has properties similar to those of PFS made by the conventional method. Importantly, the microwave method reduces the reaction time from the 500 min of the conventional method to less than 20 min, significantly reducing the amount of energy consumed and the expense of production.

Key Words: Polyferric sulfate; Microwave; ORP; Hydrolysis parameter B^*; Salicylic acid.

INTRODUCTION

Polyferric sulfate (PFS) is a pre-polymerized iron-type coagulant developed in the 1980s. Recent studies have shown that PFS is effective in the coagulation treatment process.[1] In comparison with conventional coagulants, PFS is highly efficient, less corrosive, and has a wider optimum pH range.[2–5] Coagulant production expenses can be reduced by a reduction in recycling material since the production of polyferric sulfate uses highly concentrated liquid sulfuric acid waste as a raw material.[6,7] Polyferric sulfate contains a range of preformed hydrolysis species, $[Fe(OH)_x]^{(3-x)+}$ and $[Fe_m(OH)_x]_n^{(3m-x)n+}$ of the iron (III) ion, including $[Fe_2 (OH)_3]^{3+}$, $[Fe_2 (OH)_3]_{26}^{+}$, and $[Fe_8 (OH)_{20}]^{4+}$ among others. Therefore, the polymers typically carry a high cationic charge, such that their surface activity and charge neutralizing capacity make them more competitive than conventional coagulants in terms of coagulation effect.[8] PFS production methods, especially the low-temperature method frequently applied by industry, all suffer the disadvantage of high energy and time consumption, since they involve heating (50 ~ 70°C) ferrous sulfate solid in sulfuric acid solutions that contain an oxidant and catalyst and require long reaction times for oxidation and polymerization. Consequently, this research aims progressively to apply closed microwave radiation to increase reaction efficiency and reduce oxidant and catalyst dosages to reduce production expenses and shorten the reaction time. The study also determines whether conventional and microwave methods differ in form and application.

PFS product characteristics were evaluated by the following analysis. First, according to our previous study,[6] the Oxidation Reduction Potential (ORP) of a PFS solution indicates the ratio of the substance that is being oxidized to that of its oxidized form. Thus, the ORP value was used to evaluate the degree of oxidation of iron (II) sulfate to iron (III) sulfate in the preparation process since OPR values provide stable and complete surveillance of PFS production efficacy. Second, characteristic comparisons such as of viscosity, extent of hydrolysis (B* value), salicylic acid reaction ability, and coagulation efficiency can be used to understand the hydrolysis species of PFS that was prepared using the microwave method.

EXPERIMENTAL PROCEDURES

1. PFS Preparation

Microwave method. Figure 1 shows the microwave reactor for PFS preparation. Solid ferrous sulfate (2.89 M) and $NaClO_3$ (0.375 M) were added to liquid sulfuric acid of designated concentrations to make Reagent 1, and various concentrations of HNO_3 solution were added as oxidants to make Reagent 2. A Teflon tube (10 m long and capacity 300 mL) in the microwave equipment (800 Watt) acted as a reactor for oxidation and polymerization, and reaction times were controlled by the flow rates of Reagent 1 and Reagent 2. Reaction pressure, set at 150 psi, was controlled by a back-pressure controller and after microwave heating the solution temperature was about 378 to 383°K. The resulting product was a dark-brown viscous material with the following characteristics Fe (III) $>$ 150 mg/L; Fe (II) $<$ 1 mg/L; r values $= 0.4$ ($r = [OH^-]/[Fe]$).

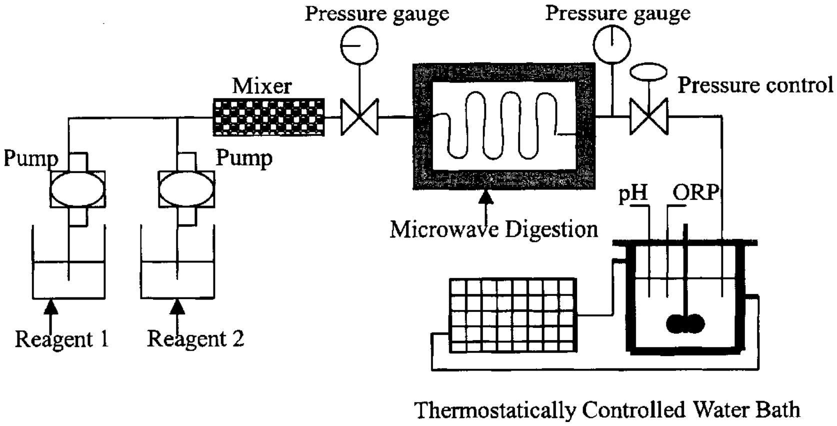


Figure 1. Experimental microwave apparatus for PFS production.

Conventional method. Solid ferrous sulfate (2.89 M) was added to liquid sulfuric acid at designated concentrations. The mole ratio of sulfuric acid to ferrous sulfate was maintained at 0.3:1. Then, $NaClO_3$ (40 g/L) and $NaNO_2$ (16.7 g/L hr) were added as an oxidant and catalyst, respectively. Pure oxygen was also added to accelerate the oxidation of ferrous sulfate. The temperature was maintained between 353°K, as recommended by Mikami,[1] to increase the solubility of the solid ferrous sulfate and accelerate the reaction. Finally, the product was aged at 323°K for 5 hours to stabilize the PFS.

2. Analysis of PFS Characteristics

(1) ORP Values

The Nernst equation relates the oxidation/reduction potential (ORP) of a system to the ratio of the concentration of the substance that is being oxidized to that of the oxidant:

$$E = E^0 + (RT/nF)\ln([\mathrm{Oxide}]/[\mathrm{Red}]) \tag{1}$$

where R = gas constant, T = absolute temperature, n = the number of electrochemical gram-equivalent per-gram moles exchanged during the redox reaction, F = Faraday's constant, E^0 = the standard electrode potential, [Oxide] = the molar concentration of the oxidant, and [Red] = the molar concentration of the reducing agent. When the reducing agent of ferrous sulfate (Fe (II)) has nearly completely oxidized, *ln*([Oxide]/[Red]) approaches infinity and the E or ORP values increase abruptly.[6] Consequently, comparing ORP values can lead to the identification of the characteristics of PFS products.

(2) Viscosity

PFS viscosity was measured using a Cannon–Fenske viscometer. The times taken for the PFS solution and distilled water to pass through the viscometer (T_{pfs} and T_{slov}) were recorded and the following formula was applied to calculate viscosity values.

$$\frac{\upsilon_{pfs}}{\upsilon_{slov}} = \frac{\rho_{slov} \times T_{pfs}}{\rho_{pfs} \times T_{slov}} \tag{2}$$

where υ_{pfs} and υ_{slov} are the respective viscosities of PFS and distilled water, and ρ_{pfs} and ρ_{slov} are the respective relative densities of polyferric sulfate and distilled water.

(3) Hydrolysis Parameter, B* Value

PFS was diluted to desired concentrations (10 mg/L as Fe) with distilled water that contain $NaClO_4$ (0.01 M). After mixing, the pH values of the solutions were obtained using a digital pH meter which had been calibrated using phosphatic buffers at pH 4 and 7. B*, which represents the extent of PFS hydrolysis, was determined from the solution's pH values immediately after dilution, by applying the Tang and Stumm's formation equation, $B^* = B_H + B - A$.[9,10]

(4) Salicylic Acid Reaction Ability

2, 4, 6, 8, and 10×10^{-5} M salicylic acid were added to 900 mL of distilled water that contained 10^{-2} M $NaClO_4$. Solution pH values were then adjusted to 2 and the same dosages of $FeCl_3$ (1×10^{-4} M as Fe) were added to each solution. Stirring changed the color of the solutions to purple. The purple solutions were then analyzed for absorbance (Abs) using a UV/VIS analyzer (1 cm cell; Hitachi model U-2000) at a wavelength of 525 nm, to determine working curves for Fe-salicylic acid complexes. These working curves were then used to determine the PFS dosage required to react completely with 1×10^{-4} M salicylic acid.

(5) Jar-Tests

A six-beaker jar test was set up at room temperature for each trial. Each beaker contained 900 mL of synthetic water that was prepared using 10^{-2} M $NaClO_4$ and 0.1 mg/l γ-Al_2O_3. The turbidity of the synthetic water was 40 NTU. The coagulants were added to the beakers that contained the synthetic water and the pH values were immediately adjusted to the specified values, using NaOH or HCl. Rapid stirring at 120 rpm for 1 min was followed by flocculation for 20-min at 30 rpm, and then a 30-min settling period. Sample pH values were recorded after settling. Samples were taken from 3 cm below the water surfaces and turbidity values were measured.

RESULTS AND DISCUSSION

1. Characteristics of PFS Made by the Microwave Method

The preparation of PFS coagulant involves oxidization and aging steps. During the oxidization step, $FeSO_4$ is added as the raw material to a fixed concentration of sulfate acid, and then the oxidant is added to oxidize Fe (II) to Fe (III). The resulting products are then aged Fe (III) involving hydrolysis and polymerization. This study, addressed the differences between products obtained using PFS that was prepared using the microwave and those obtained using PFS conventionally prepared.

(1) Oxidization

Figure 2 plots the amount of Fe (II) converted to Fe (III) versus reaction times at various HNO_3 concentrations using a microwave method. The conversion rate of Fe (II) to Fe (III) is given by

$$\eta = \frac{\mathrm{Fe(II)_0} - \mathrm{Fe(II)_t}}{\mathrm{Fe(II)_0},} \tag{3}$$

where Fe $(II)_0$ is the initial concentration of Fe (II) (M), and Fe $(II)_t$ is the remaining Fe (II) concentration (M) in the mixture at time t.

Increasing the HNO_3 concentration increased the conversion rate (η) and the reaction was observed to proceed relatively rapidly since it was completed in only 200 to 500 sec results that closely agree with the stoichiometry, which holds that the extent of Fe (II) conversion is proportional to the HNO_3 concentration.

Figure 3 plots the measured Fe (II) conversion versus initial HNO_3 concentration for particular reaction times. The gradient is approximately equal to 1, suggesting that the relationship between the HNO_3 dosage and the Fe (II) conversion is 1:1. The following formula describes this relationship.

$$\mathrm{Fe^{2+} + NO_3^- + 2H^+ \longrightarrow Fe^{3+} + NO_2\uparrow + H_2O} \tag{4}$$

Figure 3 also shows that the intercept increased with the reaction time from 0.0303 at 200 sec to 0.1716 at 1000 sec. Theoretically, when no HNO_3 oxidant is added ($x = 0$), the concentration of Fe (II) should exhibit no change ($y = 0$). However, this result shows that under microwave heating, Fe (II) is converted to Fe (III) and a longer microwave reaction time corresponds to a greater degree of Fe (II) ion conversion. According to Allal's 1996 study[11] in

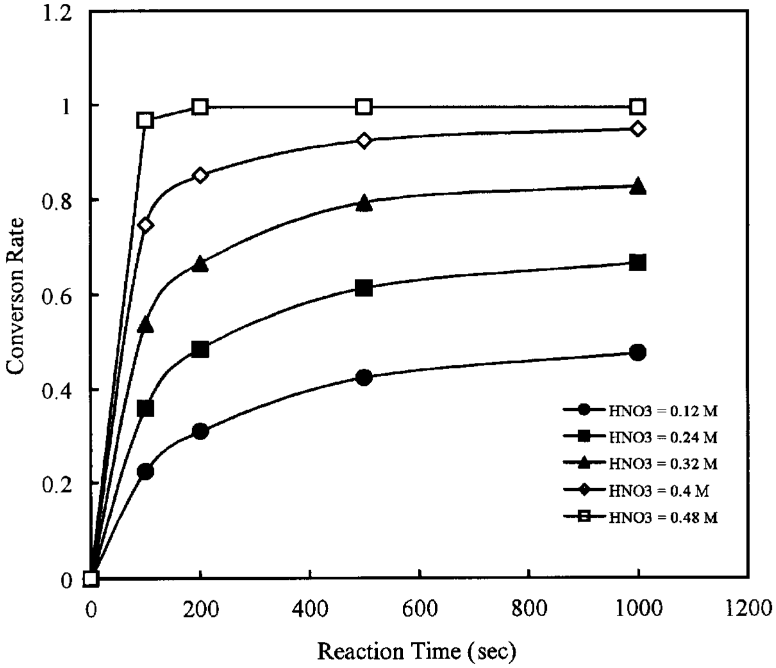


Figure 2. Influence of HNO_3 concentration on the extent of Fe(II) conversion.

which HNO_3 was used as an oxidant under ambient conditions, the reaction rate was $R = k[HNO_3][FeSO_4]$; the activation energy was 14,250 J-mole^{-1}, and at 323°K, the velocity constant, k, was 0.1 hr^{-1}. Accordingly, the relationship between the concentration of the reactant and the reaction time can be expressed as

$$\frac{1}{\mathrm{Fe(II)}_0 - [\mathrm{HNO_3}]_0} \ln \frac{[\mathrm{HNO_3}]_0 \times \mathrm{Fe(II)}_t}{\mathrm{Fe(II)}_0 \times [\mathrm{HNO_3}]_t} = kt \tag{5}$$

Thus, if the initial concentrations of Fe (II) and HNO_3 are 0.65 M and 0.48 M, respectively, and assuming a 50% conversion rate ($\mathrm{(Fe(II))}_t = 0.325$ M and $[\mathrm{HNO_3}]_t = 0.155$ M), the reaction time requirement determined by Eq. (5) is 25.7 hours at 323°K. At room temperature, the conversion rate can be predicted to be slower than at 323°K.

Figure 4 shows measured Fe (II) conversion rates with and without microwave reaction. The rate of Fe (II) conversion by HNO_3 in the presence of H_2SO_4 at room temperature and without microwave reaction, was only 0.25.

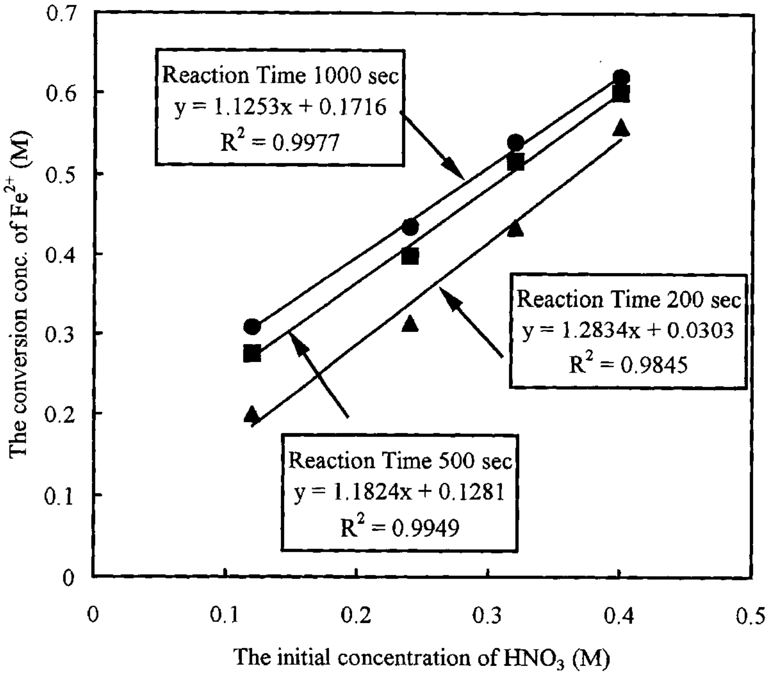


Figure 3. Relationship between HNO_3 concentration and concentration of converted Fe (II) for various reaction times.

This result suggests that H_2SO_4 acts as a catalyst of the reaction. When the H_2SO_4 concentration was reduced, the reaction rate slowed, implying that the residual HNO_3 alone could not oxidize Fe (II) without heating. Notably, in Fig. 4, after reaction time of around 100 sec, using microwave heating and the same material concentrations, the Fe (II) conversion rate reached 1. These results all demonstrate that a microwave-heated reaction not only saves much reaction time, but also increases Fe (II) conversion rates and reduces reaction expenses.

(2) Aging

After oxidation, PFS solutions must be aged to promote polymerization and the formation of stable structures. During aging, the pH values of the solutions fall because of the continuous bonding of OH^- ions onto Fe (III) ions and the release of H^+ ions into solution during hydrolysis and

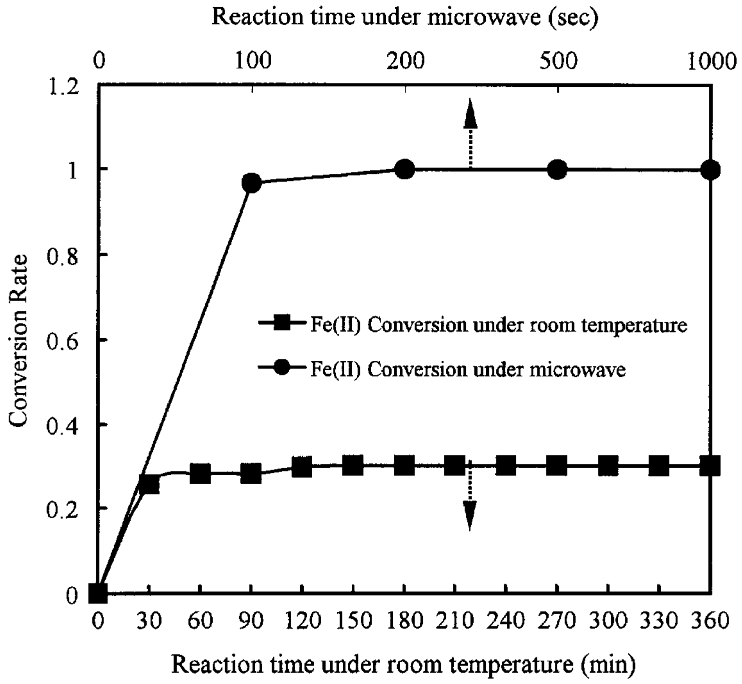
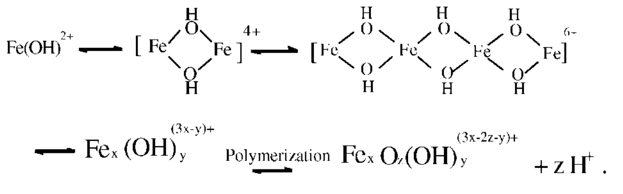


Figure 4. Effect of microwaves on oxidation rate for $[HNO_3] = 0.48\,M$.

polymerization. Tang and Stumn[9,10] presented a qualitative hydrolysis and a polymerization process scheme to elucidate the H^+ ion-release process at various stages:

$$Fe^{3+} + H_2O \rightleftarrows Fe(OH)^{2+} + H^+ \tag{6}$$



$$\longrightarrow Fe_x(OH)_y^{(3x-y)+} \xrightarrow{\text{Polymerization}} Fe_xO_z(OH)_y^{(3x-2z-y)+} + zH^+ . \tag{7}$$

As Jiang (1998)[12] stated, the conventional PFS preparation method requires that after oxidation, solutions be maintained at temperatures above 323°K for a 2-hour aging period to provide the energy required for aging. Consequently,

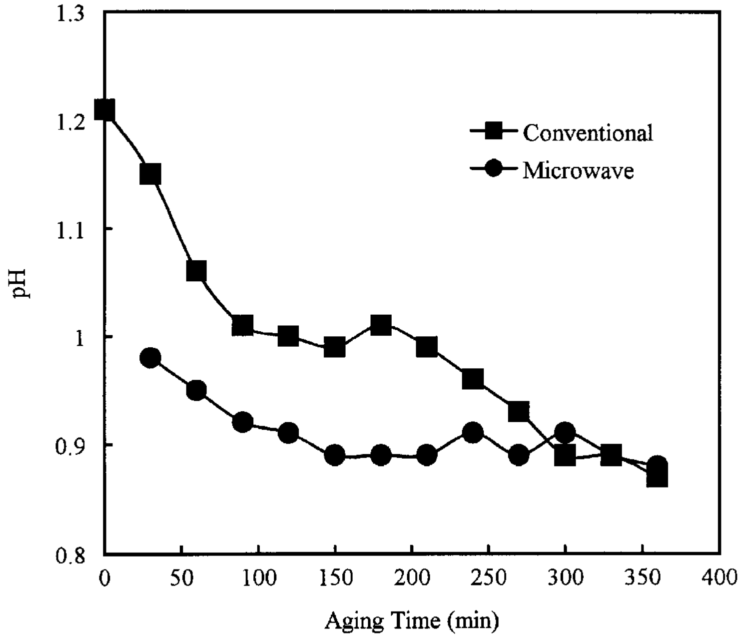


Figure 5. pH values during PFS aging.

PFS produced via both conventional and microwave methods were subjected to the same aging process in our study. Figure 5 presents the differences in pH profiles during aging at 323°K. The pH profiles of microwave PFS products remained more stable and uniform than those of conventional PFS products, perhaps because the high temperature induced by microwave radiation accelerated the rate of Fe (III) ion hydrolysis. Therefore, the Fe (III) ions completed hydrolysis and polymerization during microwave oxidation, and consequently, pH profiles no longer changed greatly during aging. Using PFS produced by the microwave method may thus enable significant reduction or complete elimination of the aging process, which would reduce time and heating expenses. Therefore, the time required to produce PFS may be reduced to less than 20 min using a microwave method.

2. Evaluating the Characteristics of PFS

In our previous study,[6] ORP values (mv) were used to evaluate the degree of oxidation of iron (II) to iron (III) during the preparation of PFS.

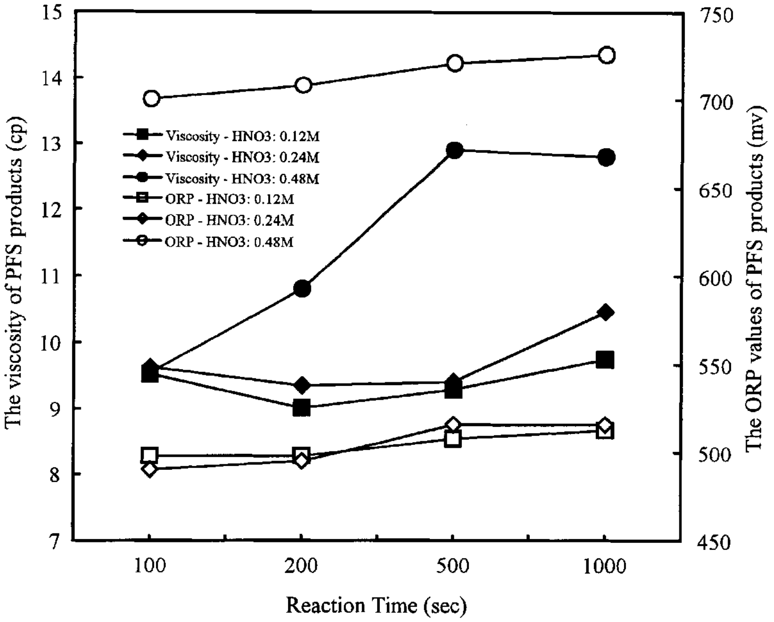


Figure 6. Comparisons of PFS product viscosity and variations in ORP value for preparations made with different concentrations of HNO_3.

Accordingly, when the results shown in Fig. 6 are observed, no increase in ORP values for PFS products formed using HNO_3 doses from 0.12 M to 0.24 M was evident, implying that this HNO_3 concentration range was insufficient to oxidize all Fe (II) to Fe (III) even when the reaction time was increased to 1000 sec. However, comparing the conversion rates in Fig. 2 with the ORP values in Fig. 6 shows that Fe (II) conversion was complete when the HNO_3 concentration reached the adequate level of 0.48 M, and that the ORP value increased to 700 mv even when the reaction period was only 100 sec.

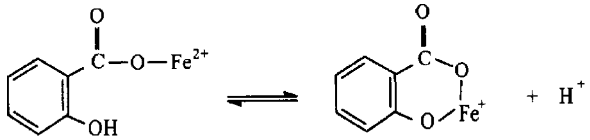
An important factor that determines the extent of polymerization of PFS is the ratio, $[SO_4^{2-}]_T/[Fe]_T$, or the *r* value ($r = [OH^-]_T/[Fe]_T = n/2$, obtained from the PFS formula $Fe_2(OH)_n(SO_4)_{3-n/2}$. The formula gives the number of OH^- ions to which each iron ion bonds during aging or hydrolysis. The number is related to the dose of sulfuric acid used in PFS preparation. Jiang and Graham[12] found that PFS prepared with lower $[SO_4^{2-}]_T/[Fe]_T$ ratios (higher *r* values) includes higher proportions of polymeric and precipitation species, reducing the PFS's stability. PFS products that contain higher percentages of small $Fe(OH)_3$ precipitate species are believed to have a lower viscosity than those with less of such species. Accordingly, an optimum formulation of PFS, with an $[SO_4{}^{2-}]_T/[Fe]_T$ ratio of $1.3 \sim 1.4$ ($0.2 < \gamma < 0.4$), represents a balance between polymerization and product stability.[12] Consequently, the optimum $[SO_4{}^{2-}]_T/[Fe]_T$ ratio was set at 1.3 ($\gamma = 0.4$) for PFS prepared using the microwave method in the experiments considered here, and the degree of PFS polymerization and stability were estimated from its viscosity. The results in Figs. 2 and 6 show that, when the HNO_3 dose did not suffice to oxidize all Fe (II) ions, the PFS product viscosity was lower, clearly implying that residual Fe (II) ions led to reduced PFS product stability. The curve for 0.48 M HNO_3 in Fig. 2 shows that even with only 100 sec of reaction time, the oxidant was sufficient to convert 99% of the Fe (II) to Fe (III). However, comparing this result with Fig. 6 shows that although the viscosities for various reaction times were not similar, they still increased with reaction time. Jiang and Graham[12] indicated that increasing the aging temperature or the aging time increased PFS product polymerization. Thus, the 0.48 M HNO_3 curve in Fig. 6 shows that product viscosity became smoother after 500 sec of reaction time, revealing that this reaction time suffices for oxidation and polymerization. However, after 3 months, stored PFS products made with 1000 sec of reaction time showed viscosity changes from their original 13.58 cp to 9.32 cp. In contrast, after 3 months, the viscosity of stored PFS products made with 500 sec of reaction time still had remained at approximately about 12.9 cp, and the products were stable, exhibiting no precipitation. This result also shows that PFS products become unstable and precipitate after oxidation over a long period using microwaves. Comparing the $PFS_{r=0.4}$ viscosity of PFS produced by the microwave method (0.48 M HNO_3, 500 sec.) with that of PFS produced by the conventional method $PFS_{r=0.4}$ (viscosity 13.13 cp, obtained in our previous study[6]) shows that $PFS_{r=0.4}$ prepared using these two different methods has similar viscosity characteristics. Accordingly, the results of the experiment described above establish that, at the same γ values, PFS made by the microwave method has pH, ORP, and viscosity characteristics similar to those of PFS made by the conventional method.

3. Evaluating the Reactivity of PFS

(1) Salicylic Acid Reactivity

Our previous study[13] demonstrated that, under lower pH conditions (pH < 4), purple complexes were formed by Fe (III) ions and salicylic acid.

The complex reaction is



(8)

The reaction of salicylic acid with monomer Fe (III) ion initially forms a soluble complex with 1/1 stoichiometry.[13,14] Conversely, as the complex percentage curve shown in Fig. 7 clearly indicates that every salicylic acid molecule is bonded to more than one Fe ion. PFS, being a preformed hydrolyzing coagulant that cannot bond with salicylic acid, follows the mechanism described in Eq. (8) in one-to-one stoichiometry. Figure 7 presents the difference between salicylic acid reactivity for PFS products prepared using the conventional method ($\gamma = 0$ and 0.4) and that for PFS products prepared using the microwave method. For example, $PFS_{\gamma=0}$ and $PFS_{\gamma=0.4}$ require 2×10^{-4} M and 3×10^{-4} M iron ions, respectively, to realize 80% of their salicylic acid reactivity. Thus, PFS possessing more polymeric species

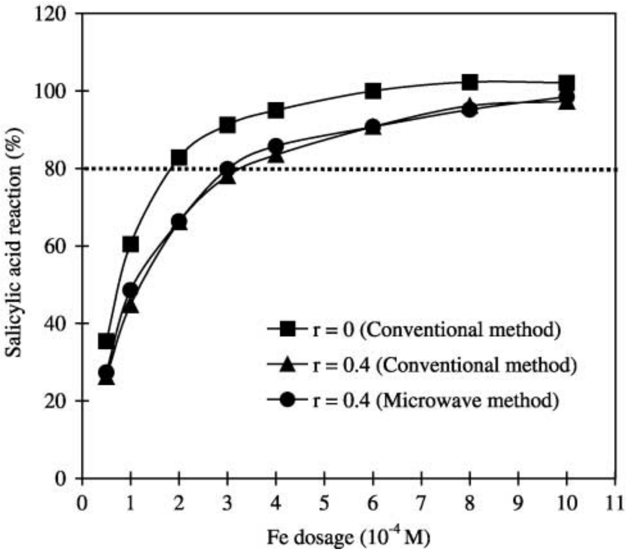


Figure 7. Salicylic acid (1×10^{-4} M) complex percentage against concentration of different PFS products.

requires more ferric ions to react with each molecule of salicylic acid. Consequently, the extent of PFS polymerization can be estimated by measuring its salicylic acid reactivity. Figure 7 also reveals that the curves for $PFS_{\gamma=0.4}$ prepared by the conventional and microwave methods (0.48 M HNO_3, 500 sec) are very similar, indicating that the degree of polymerization of microwave $PFS_{\gamma=0.4}$ is similar to that of conventionally made PFS.

(2) Hydrolysis Parameter B*

Tang[9,10] applied a formation function to characterize the extent of Fe (III) hydrolysis and polymerization:

$$B^* = B_H + B - A, \tag{9}$$

in which,

$$B^* = [OH^-]_{bond}/[Fe]_T$$

$$B_H = [H^+]/[Fe]_T = 10^{-pH}/[Fe]_T$$

$$B = [OH^-]_{add}/[Fe]_T$$

$$A = [H^+]_{add}/[Fe]_T$$

B* is the number of OH^- ions to which each ferric ion bonds during hydrolysis; B_H is the initial ratio, which is the counterpart of the concentration of the free H^+ in the iron solution and can be determined from the pH value, and $[OH^-]_{add}$ and $[H^+]_{add}$ are, respectively, the concentrations of strong base and acid added. The formula for PFS, $Fe_2(OH)_n(SO_4)_{3-n/2}$, reveals that it is a pre-hydrolytic iron salt. When the same amounts of various types of PFS were added to distilled water that contained $NaClO_4$ (0.01 M), the B* values were inversely proportional to the *r* values ($r = [OH^-]/[Fe] = n/2$, obtained from the formula, $Fe_2(OH)_n(SO_4)_{3-n/2}$, and relating to the sulfuric acid dosage used in PFS preparation). Thus, PFS with a lower $[SO_4^{2-}]_T/[Fe]_T$ molar ratio (or higher *r* value) requires fewer OH^- ions during hydrolysis yielding lower B* values. Consequently, in theory, PFS preparations with the same *r* values are similar in terms of product $[OH^-]/[Fe]$ ratios, so their B* values should be the same. In this study, B* tests of $PFS_{r=0.4}$ products prepared by the microwave (0.48 M HNO_3, 500 sec) and conventional methods showed that their B* values were 2.19 and 2.10, respectively.[8] This result indicates that the [OH$^-$]/[Fe] ratios of the products were similar and their extents polymerization were also close.

(3) Coagulation Efficiency

The ability of the product to treat water turbidity was measured to evaluate its efficiency as a coagulant. Accordingly, $PFS_{r=0.4}$ products prepared by the microwave (0.48 M HNO_3, 500 sec) and conventional methods[6] were added to synthetic water that contained γ-Al_2O_3 (0.1 mg/l), and whose initial turbidity was 40 NTU. Figure 8 presents the jar test measurements. Comparing the treatment efficiency profiles shows that coagulant prepared by the microwave method yielded results similar to those obtained using coagulant prepared by the conventional method, since similar turbidity residual was obtained at various coagulation dosages. Consequently,

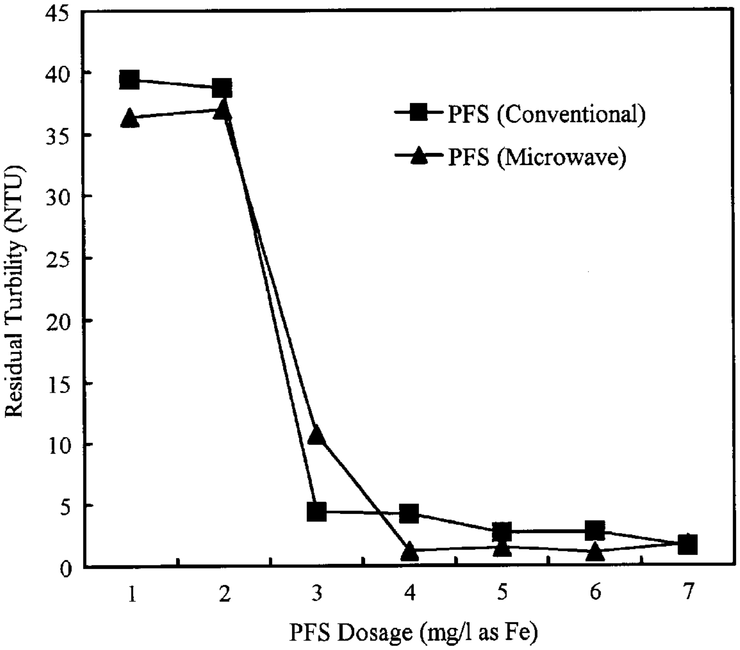


Figure 8. Percentage of turbidity removed after jar mixing and settling for various coagulant dosages (pH 5.5).

the results detailed here strongly imply that the new microwave method is a superior choice for PFS preparation.

CONCLUSION

This study applied a microwave method to reduce the reaction time and energy consumption of the PFS production process. Experimental results indicate that Fe (II) sulfate can be efficiently oxidized to Fe (III) sulfate by microwave heating using low HNO_3 concentrations and short reaction times than required by the conventional method. Producing PFS by the microwave method may also lead to a significant reduction or elimination of the aging process, which would save time and heating expenses. Comparison of PFS product physical and chemical characteristics reveals that $PFS_{r=0.4}$ prepared by the microwave method with a nitric acid concentration of 0.48 M and a 500 sec. reaction time exhibits characteristics similar to those of $PFS_{r=0.4}$ prepared by the conventional method. These results clearly demonstrate the feasibility of the new method of producing PFS coagulant, using continuous microwave heating.

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